Study of microstructural defects in stainless steels with positron annihilation spectroscopy

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1. Introduction

Stainless steels(SS) are used mainly to structural materials of light water reactor(LWR). The structural materials are usually around reactor core, so temperature of SS increases($\geq$ 300). Temperature is important factor to determine defect density of materials. Among the methods available for estimating the defect density, transmission electron microscopy (TEM) is a power tool to investigate the dislocation structures in deformed metals and to derive parameters to describe them[1]. Positron annihilation lifetime spectroscopy (PALS) is a sensitive method for investigating open-type defects which include vacancies, vacancy agglomerates, and dislocations. The measured positron annihilation lifetimes of a sample are linked to the size of the defects and the relative intensities of each defect are related to the defect concentrations[2]. The present paper is intended to study of microstructual defects for annealed stainless steel samples after cold-worked by using PALS analysis.

2. Experimental

The material used for this study is commercial-grade stainless steel (SS) 316. The chemical compositions of SS316 are listed in Table 1. The samples were prepared by a conventional cold-working at room temperature with different percentages of a deformation. They were cold worked by 0%, 20%, 40% and 80%. And thermal treatments at 300 ℃ and 700 ℃ for 6hr in vacuum followed by furnace cooling(FC).

Table 1. Chemical compositions of SS316 [wt%]

Cr	Ni	C	Mn	Si	P	S	Mo	Fe
16	12	0.08	2	1	0.045	0.03	2.5	Bal

2.1 Microstructure Analysis

The annealed microstructures were studied with optical microscopy (OM) and transmission electron microscopy (TEM) techniques. The OM images were taken after electro-chemical polishing in the electrolyte consisted of 40ml H_2SO_4 + 60ml H_3PO_4 + 10ml H_2O + 20ml glycerin at 70°C with 1.0 A/cm^2. The TEM specimens were prepared by twin-jet electro-chemical thinning (TenuPol-5, Struers) with the electrolyte consisted of 700ml CH_3OH + 200ml butylcellosolve + 100ml $HClO_4$ at about -35°C. The microstructures of the specimens were observed by using a field emission transmission electron microscopy(FE-TEM) with JEM-2010F (JEOL).

2.2 PALS Measurement

The PALS measurements were performed at room temperature by means of a fast-fast coincidence timing spectrometer. We employed a ^{22}Na β^+-source of about 1 MBq and collected more than one million counts for each test. The positron lifetime can be measured by detecting the time difference between the birth γ-radiation of the ^{22}Na β^+-source and one of the annihilation γ-quanta with energy of 511 keV. The scheme of the positron lifetime measurement is shown in Fig. 1. The time resolution of the system is 260 ps in full width at half maximum. The positron lifetime data was analyzed by subtracting the source components and background. All the spectra were decomposed into two lifetime components by using the PALSfit program[3].

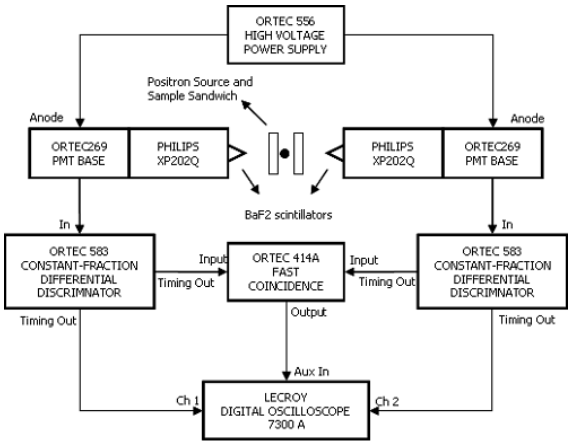


Fig. 1. Scheme of the positron annihilation lifetime spectroscopy.

When there are two types of defects(vacancies and dislocations) in the lattice, we have to apply a three-component fit to PAL spectra. In this case, it is probable to obtain poor statistics due to the uncertainty about the analyzed lifetimes[2]. Accordingly, we decompose the spectra using only two lifetimes with fitting parameters τ_1(short), τ_2(long), and I_2. In interpreting the lifetime data, the τ_1 component was assumed to be weighted average of annihilation in the dominant bulk as well as a shorter lifetime trap, whereas the τ_2 component is related to the relatively big-sized traps such as vacancy clusters. From the analysis of the standard trapping model[4], we can

derive the dislocation density with proper information on the positron trapping coefficients.

3. Results

All of the specimens prepared by annealing at 300°C for 6hr in vacuum still hold the microstructure of cold worked structure and this microstructure have the elongated area having got a recrystallization process since it was not enough to complete the recrystallization at this condition.

In the case of 700°C shown in Fig. 2, the recrystallization of the 20% cold-worked specimen was more completed than the 40% specimen, because the latter have more defects then the former. The strain-induced grains during annealing may potentially grow and become the nuclei for recrystallization. A completed grain starts to grow and consumes neighbouring grains having high-density dislocations. The recrystallization of the 80% specimen was completed at a small part of grains.

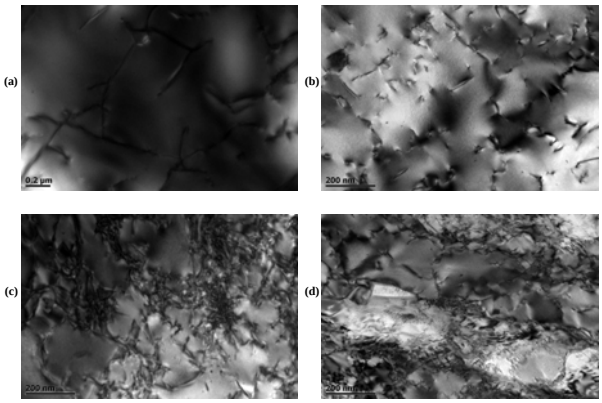
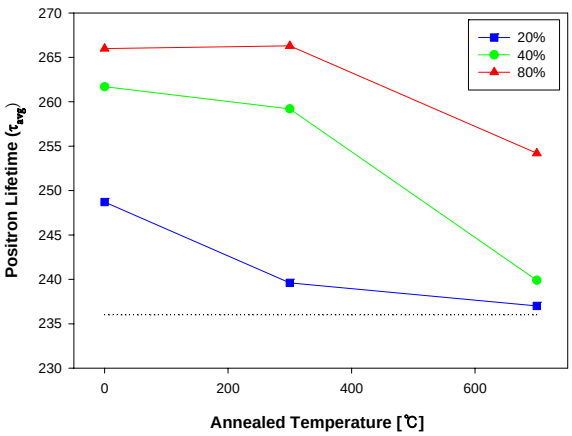


Fig. 2. TEM micrograph of SS316 (a) 0%, (b) 20%, (c) 40%, (d) 80% cold-working in 700 ℃.

The PALS lifetime data are shown in Fig. 3. Short (τ_1) and long(τ_2) annihilation lifetimes are derived from all samples using the standard trapping model (STM).



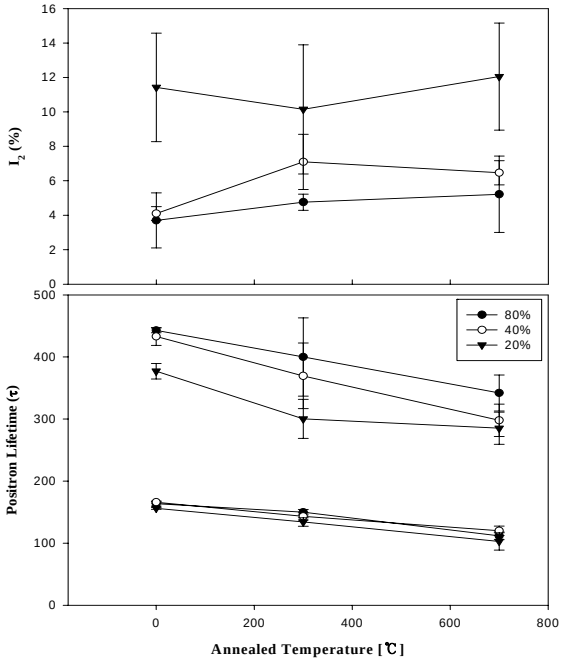


Fig. 3. PALS measured average lifetimes, relative intensities and short(τ_1) and long(τ_2) lifetimes at the different annealing temperature of SS316

At 700℃, the measured lifetime τ_1 has a range from 103 to 111 ps, which is like a characteristic value of defect-free Fe (~110 ps).

The long lifetime(τ_2) decreases as temperature increases, while its intensity increases slightly(~2%). In other word, the size of open-volume decreases, while its amount slightly increases. It is believed that the tangled defects made up of big-sized vacancy clusters and dislocations are more easily dissociated at the higher temperature.

4. Conclusions

Two different methods, TEM investigation and PALS analysis, were applied to estimate the change of defects density for both cold-worked and annealed SS316 at the different temperature. It is found that two methods are useful in investigating behavior of defect densities and are complementary each other.

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